

# Solid-On-Liquid Technology for Suspended Microchannel Fabrication

Antoine Aupée

Fall semester 2015



ÉCOLE POLYTECHNIQUE  
FÉDÉRALE DE LAUSANNE

## **Abstract**

This report summarizes the work of a Microengineering's student semester project realised in the GR-LVT laboratory of the Swiss Federal Institute of Technology in Lausanne. This work was supervised by Dr. Villanueva and his doctoral assistant A. De Pastina. The project was to find a way to engineer Parylene C microchannels on top of LS-SiN cantilevers.

Professor : Guillermo Villanueva  
PhD Assistant : Annalisa De Pastina

# Contents

<b>1</b>	<b>Introduction</b>	<b>3</b>
<b>2</b>	<b>Project Presentation and initial work in La Chaux-de-Fonds</b>	<b>4</b>
2.1	Project specifications . . . . .	4
2.2	La Chaux-De-Fonds experiments . . . . .	4
2.2.1	Parylene Deposition . . . . .	4
2.2.2	Laser digging . . . . .	6
2.2.3	Silane coating . . . . .	7
<b>3</b>	<b>First Process: FOTS Deposition</b>	<b>8</b>
3.1	Presentation and Process Flow . . . . .	8
3.1.1	Presentation . . . . .	8
3.1.2	Process Flow . . . . .	8
3.2	FOTS deposition process and Results . . . . .	9
3.2.1	First try . . . . .	9
3.2.2	Second try with adjusted parameters . . . . .	9
<b>4</b>	<b>Second Process: Photo-Resist as Sacrificial Material</b>	<b>11</b>
4.1	Presentation and Process Flow . . . . .	11
4.1.1	Presentation . . . . .	11
4.1.2	Process Flow . . . . .	11
4.2	Design of the channels and inlets via Photolithography . . . . .	12
4.2.1	Design . . . . .	12
4.2.2	Dosetests . . . . .	12
4.3	Encapsulation of the microchannels with parylene C . . . . .	15
4.4	Opening of the inlets . . . . .	15
4.4.1	Second lithography aligned on the initial design . . . . .	15
4.4.2	Dry etching of the parylene and the LS-SiN . . . . .	16
4.5	Cantilever release, etching of the Silicon . . . . .	18
4.5.1	Wet etching . . . . .	18
4.5.2	Dry etching . . . . .	19
4.6	Emptying the channels, etching of the Photo-Resist . . . . .	21
4.6.1	Selection of the best solvent . . . . .	21
4.6.2	Determination of the etching parameters . . . . .	22
<b>5</b>	<b>Liquid circulation test</b>	<b>24</b>
<b>6</b>	<b>Results</b>	<b>25</b>
<b>7</b>	<b>Conclusion &amp; Outlook</b>	<b>26</b>



# 1 Introduction

Last fall, I have conducted several experiments in La Chaux-De-Fonds as well as in the EPFL cleanroom in order to try to inspect the possibility of suspended microchannel fabrication.

This report will therefore go through the different experiments conducted on cantilevers.

The cantilevers described in this project are a new type of bio-sensor. Originally, properties of liquids were deduced by the oscillation of thin cantilevers plunged in it. These new bio-sensors are working on the same principle (oscillating cantilevers) but this time the liquid flows inside of them and the whole cantilever is contained in a vacuum in order to have the most precise reading on the oscillating frequency.

This new type of bio-sensor has already been fabricated but the processes used are quite long and require dangerous chemicals (the emptying of the channels are done with KOH for 12 hours). This project will help determine if the use of Parylene C (the original material of the Solid on Liquid Technology) can help reduce the time needed and facilitate the whole fabrication process.

More precisely, this report will cover two different processes. The first one based on a chemical trapping solution involving the use of a hydrophobic surface treatment and the second one, adjusted after the first experiments, based on the use of photoresist as a sacrificial material inside the microchannels.

The results described in this project seeks to help the development of future processes in order to reduce once more, the time and complications involved in the fabrication of the sensors.

## 2 Project Presentation and initial work in La Chaux-de-Fonds

### 2.1 Project specifications

As explained in the introduction, the goal is to find a process to produce microchannels on top of a suspended cantilever. These cantilevers are made of a  $100\text{nm}$  thick layer of Low Stress Silicon Nitride (LS-SiN), are  $500\mu\text{m}$  long and  $40\mu\text{m}$  wide in order to be able to arrange two  $10\mu\text{m}$  wide channels on top of it.

These designed channels of liquid are coated with a thin layer of Parylene C (about  $2\mu\text{m}$ ). Finally, the channels on the cantilevers lead to large holes ( $300\mu\text{m}$ ) which will be used as inlet/outlet in order to control the liquid flow.

All these specifications can be seen on Figure 1.

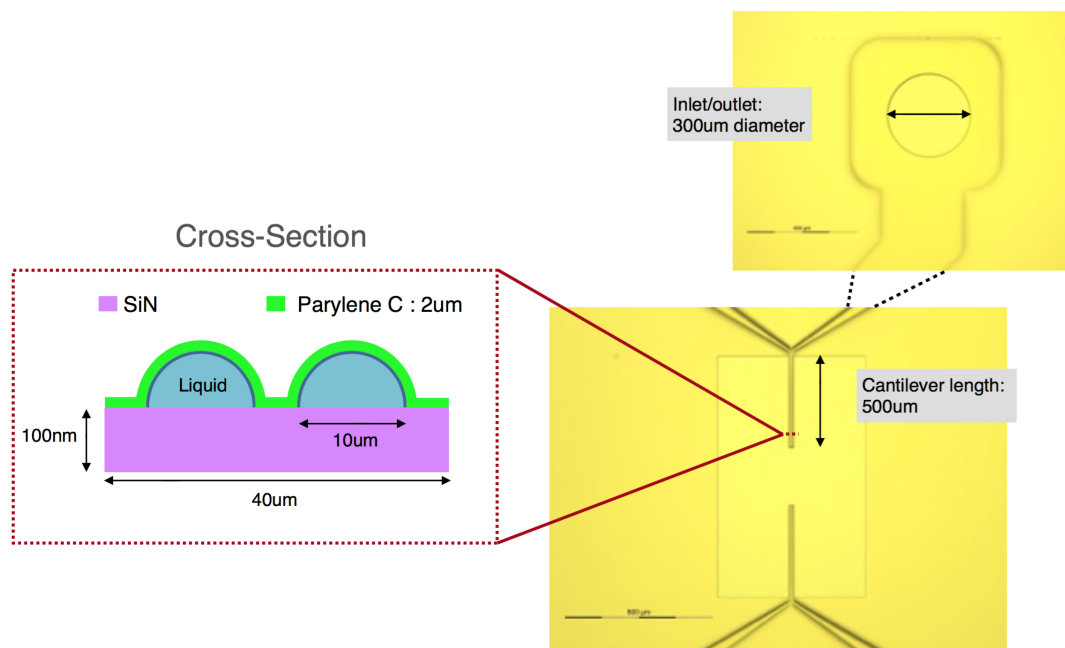


Figure 1: Design Specifications

### 2.2 La Chaux-De-Fonds experiments

Prior to the beginning of the semester, I had the opportunity to spend a week at La Chaux-De-Fonds in the Haute Ecole Arc Ingénierie (HES-SO) laboratory. In this context, I have performed several experiments to measure the adhesion between parylene and LS-SiN as well as the coating of liquids under different thicknesses of parylene.

#### 2.2.1 Parylene Deposition

##### Technology

Solid-On-Liquid Technology describes the coating of a liquid with a solid layer of parylene C. This is made possible by two major things, the high conformity of parylene C allows a complete coverage of the liquid and the lack of stress inside the parylene after its deposition prevents the liquid from moving so that the covered liquids will keep its initial shape. It is a LPCVD (Low Pressure Chemical Vapor Deposition) process where the deposition is done at Room temperature, but the covered liquid needs to be adequate in order to subsist low pressure.

Parylene is the name of a polymer family commonly used to encapsulate device with a thin polymer layer. In my case, I am using parylene C, which is better for its encapsulation and isolation properties. The molecule can be seen on Figure 2.

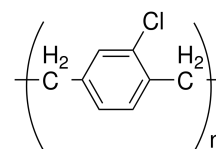


Figure 2: Parylene C molecule

To cover something with parylene, a really precise process needs to be followed in a machine devoted to it. It can be divided into three steps[2]:

1. Vaporization: The parylene dimers (white powder) are vaporized around 150°C at low pressure (1.0 Torr) and brought to the second chamber.
2. Pyrolysis: In this second chamber, the dimers are cleaved into monomers by heating them at 680°C.
3. Deposition: These monomers enter the deposition chamber and polymerizes on our substrates at room temperature (25°C).

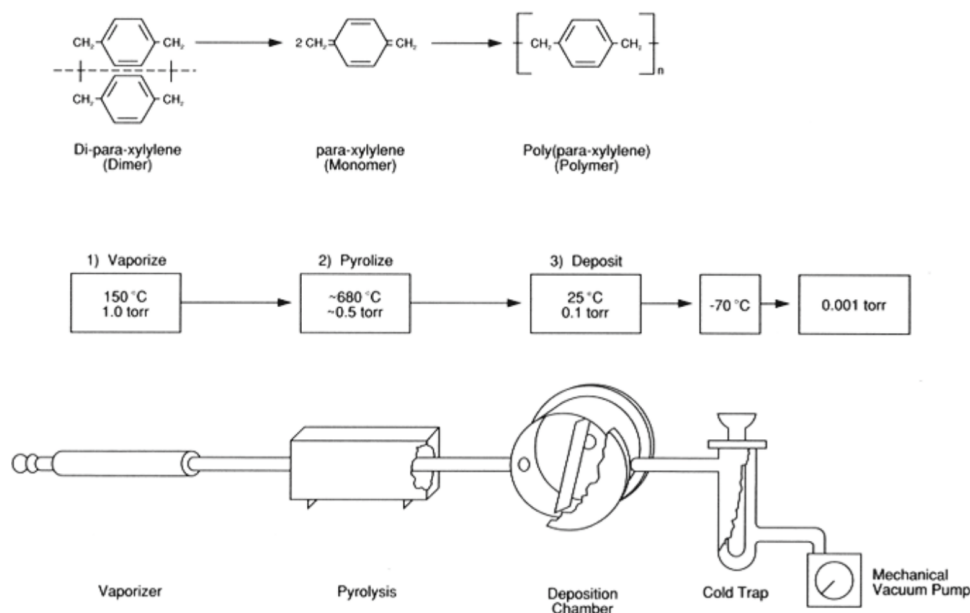


Figure 3: Parylene Deposition Process

## Experiments

The different experiences allowed me to see the correct adhesion between parylene C and LS-SiN also the good encapsulation of liquids underneath a thin layer (between 2 and 5 microns).

All the experiments were done on the Comelec machine available at La Chaux-De-Fonds laboratory (Figure 4). The layer thickness being completely proportional to the mass of parylene powder inserted in the machine, it is really easy to control the final thickness of parylene.



Figure 4: Comelec Parylene coating machine at La Chaux-De-Fonds

### 2.2.2 Laser digging

To better design the microchannels, one of the first idea was to create a physical trapping of the liquid using parylene C. It would consist of a layer of parylene (around 10 microns), then the microchannels are drawn using a laser (femto laser). The trenches created are filled with liquid and closed with a second layer of parylene (see Figure 5).

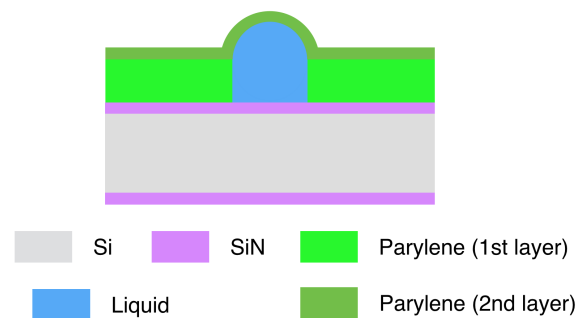
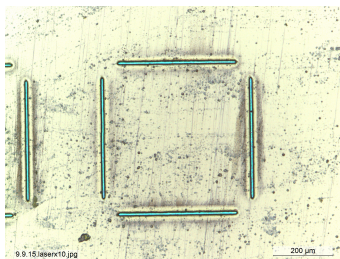


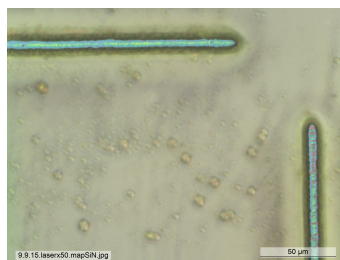
Figure 5: Cross section of a microchannel using physical trapping

The main problem encountered with this solution is the precision of the laser cut, as seen on the Figure 6, the cut width is pretty uniform but the depth is unstable. Especially on Figure 6a, the bottom of the cut is not uniform so we cannot have any certitude about the remaining thickness of Silicon Nitride underneath the Parylene.

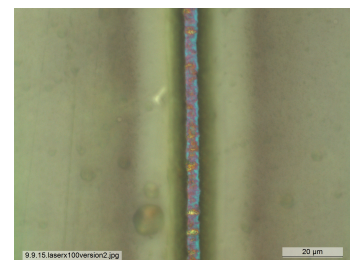
The LS-SiN layer needs to be unaltered in the process, so this solution does not seem preferable.



(a) Example of vertical and horizontal cuts



(b) Zoom of the cuts



(c) Zoom of the bottom of the cuts

Figure 6: Laser cuts of Parylene on top of LS-SiN

### 2.2.3 Silane coating

As Physical trapping by laser cuts into parylene does not suit our needs, the second experiment is try a chemical trapping. As seen on Figure 7, the goal was to put a hydrophobic surface treatment everywhere on the substrate except where the channels would be. In our case we used the silane A174 which make silicon hydrophobic. Its efficacy on LS-SiN needs to be checked.

To this end, we tried to coat LS-SiN using two methods, the first by evaporation of a liquid A174 solution inside a vacuum chamber, the second one with a liquid solution in which to dip the wafer (1/3 A174, 1/3 IPA, 1/3  $H_2O$  for 12 hours).

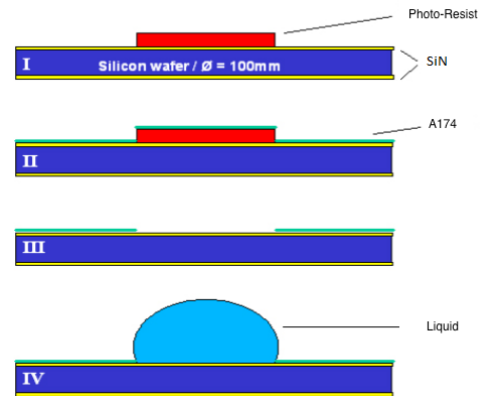


Figure 7: Silane A174 coating process

Unfortunately, the results with both methods are not good. No difference can be made in terms of hydrophobicity between the wafers with or without A174 (coated by evaporation or solution). It seems that the process for A174 coating of LS-SiN is different than for Silicon hence the bad results.

### 3 First Process: FOTS Deposition

Back to the EPFL, I got to work on two main processes described in the two following sections.

#### 3.1 Presentation and Process Flow

##### 3.1.1 Presentation

This idea is the same as the one experimented in La Chaux-de-Fonds, chemically trapping the liquid where we want and cover it with parylene to create the channels. The difference being in the chemical used, FOTS instead of A174

FOTS is also a silane, it stands for Trichloro(1H,1H,2H,2H-perfluorooctyl)silane and its role is to make the substrate's surface hydrophobic.

##### 3.1.2 Process Flow

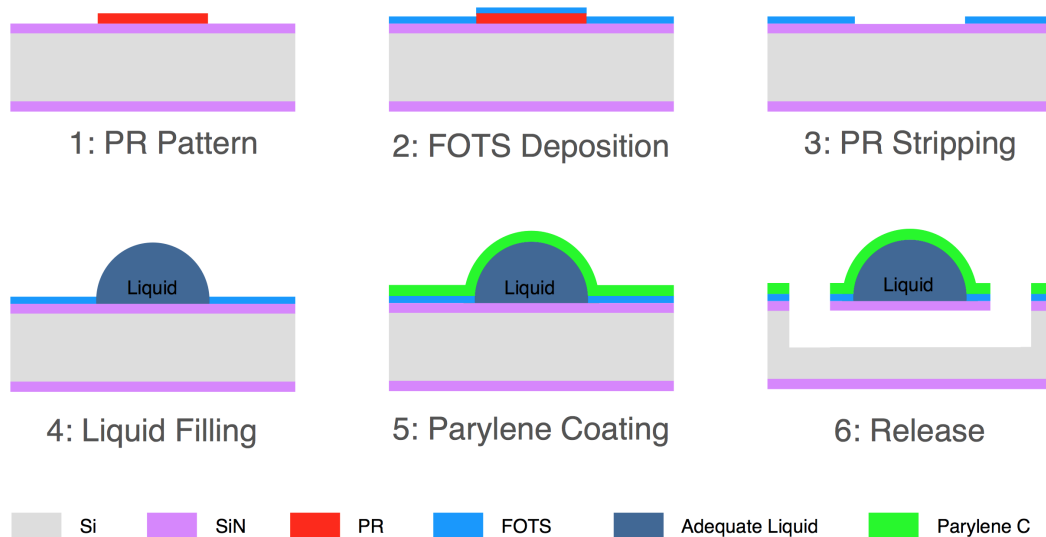


Figure 8: FOTS Deposition Process Flow

As we see on Figure 8, this process is divided in six major steps detailed here:

- 1. PR Pattern:** We take a Silicon wafer coated with a 100nm thick layer of Low Stress Silicon Nitride (LS-SiN) and we pattern the shape of our microchannels as well as inlet/outlet via photolithography (cf. Section 4.2.1).
- 2. FOTS Deposition:** After the pattern, we cover everything with FOTS by evaporation of a liquid solution.
- 3. PR Stripping:** We remove all of the Photo-Resist in order to reveal the channels under the FOTS layer.
- 4. Liquid filling:** For this step, we need to fill the defined channels with an adequate liquid. Adequate because it must sustain the low pressure of the parylene coating and with enough precision to have a uniform volume along these 500 $\mu$ m long and 10 $\mu$ m wide channels.

5. **Parylene coating:** The next step is very delicate, we need to take the wafer inside the channels in the Parylene coating machine and do the whole coating process with liquid inside the patterned area.
6. **Release:** After the liquid's encapsulation with parylene, we need to the "cantilever" from the silicon wafer. It means etching the layers of Parylene/FOTS/SiN/Si vertically around the cantilevers and then etch the silicon underneath the narrow channels without damaging them.

## 3.2 FOTS deposition process and Results

As there was multiple steps that had to be validated, I began with the FOTS deposition itself. From the literature, we know that a FOTS surface treatment onto Silicon or Silicon Oxide makes it hydrophobic with a water contact angle of  $110^\circ$ . My first job is to check if the same results with FOTS onto Low Stress Silicon Nitride are obtainable.

### 3.2.1 First try

The procedure of such a surface treatment is a standard Molecular Vapor Deposition. It means we put a little bit of FOTS liquid solution along with the wafer inside a vacuum bell; We turn on the vacuum and after 45 minutes, the FOTS is evaporated onto the wafer (cf Figure 9).

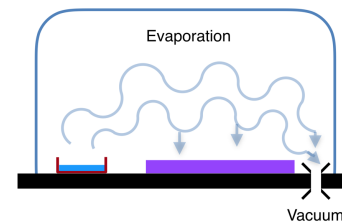


Figure 9: FOTS Deposition Method

Contact Angle	Silicon	Silicon Oxide	Silicon Nitride
Before	$24^\circ[1]$	$15^\circ[3]$	$49^\circ$
After 45mins	$110^\circ$	$110^\circ$	$35^\circ\text{-}57^\circ$

As seen in the Table, the results were not conclusive. We were expecting an hydrophobic surface and the contact angle was rather low. It was not consistent on the whole wafer, some regions were around  $35^\circ$  (lower than before the treatment) while other were around  $57^\circ$ .

As there were no obvious causes to these results, the experiment was done a second time.

### 3.2.2 Second try with adjusted parameters

First, as the FOTS solution didn't seem to attach well on the LS-SiN, the substrate was activated prior to its treatment. To this end, we placed the wafer in an oxygen plasma oven for two minutes at a power of 29W.

Secondly, instead of the previous 45 minutes, we let the wafer experiment run inside the vacuum bell for 4 hours and a half, after which the evaporation of liquid solution was complete (opposed to the first time where liquid solution remained in the container).

Despite these modifications, the results were not better. As the first try showed inconsistency, we tried to see if the results were dependent on the location of the contact angle measurement, but to no avail.

Figure 10 shows a top view of the contact angle results depending on the test's location on the wafer with adjusted parameters.

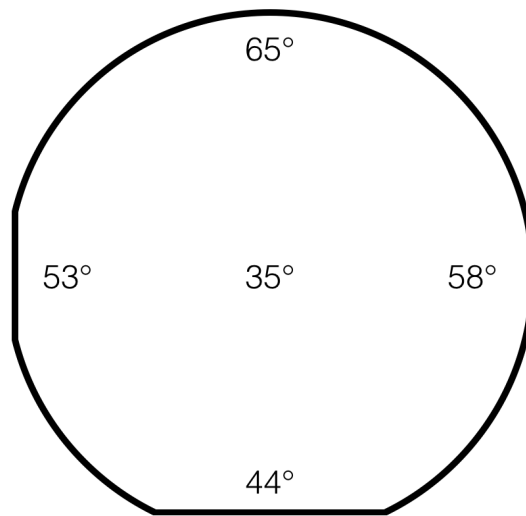


Figure 10: Top View of the contact angle measurements done right after four hours in the vacuum bell

It seems that the adhesion between LS-SiN and FOTS is not as good as the one with silicon and silicon oxide. Seeing how those first experiments went, we decided to adapt our process in order to avoid these complications. All the changes made are explained in the next section.



## 4 Second Process: Photo-Resist as Sacrificial Material

### 4.1 Presentation and Process Flow

#### 4.1.1 Presentation

As explained in the previous section, the first surface treatment done to make the wafer's surface hydrophobic was not working as supposed to. This hydrophobization was supposed to hold in place the liquid that would define the future microchannel. In order to accomplish the same thing, we had the idea to use photoresist as sacrificial material instead of liquid.

This would simplify the first steps of the process as we could directly deposit the photoresist via photolithography and we didn't need any surface treatment.

The biggest challenges of this process are to find a way to remove this photoresist once coated with parylene and release the cantilevers without damaging the microchannels.

#### 4.1.2 Process Flow

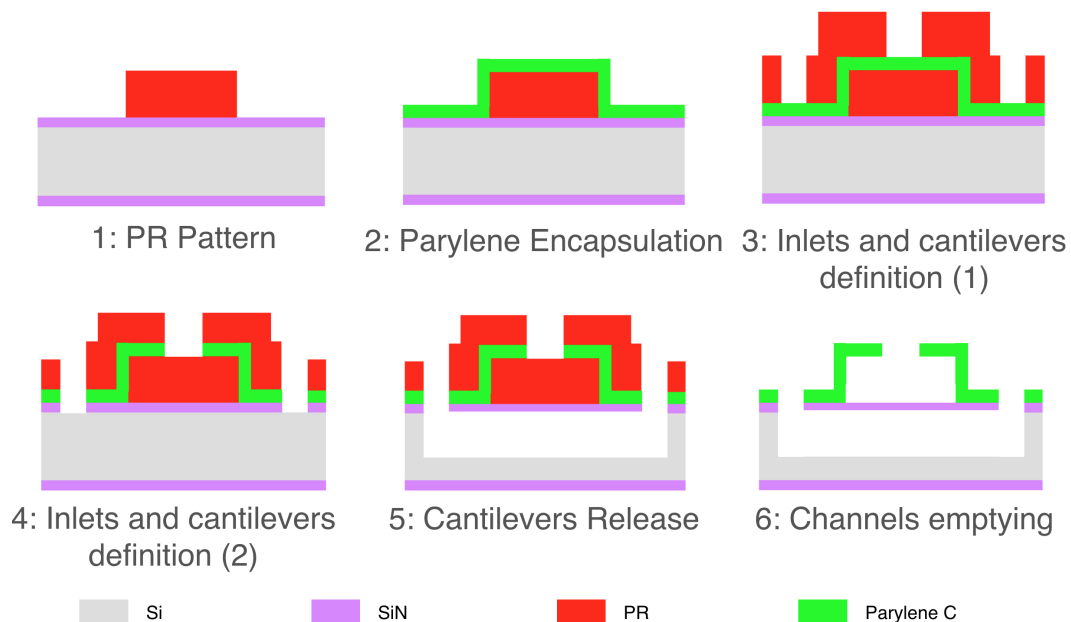


Figure 11: PR Process Flow

As we see on Figure 11, this process is divided in six major steps detailed here:

- 1. PR Pattern:** First step is the definition of the channels and inlets via photolithography, it represents the area that will be covered with parylene and removed later to let liquid flow.
- 2. Parylene Encapsulation:** After the channel's design, they are encapsulated by a parylene layer. As seen in the previous section, parylene is deposited at room temperature in a LPCVD process, so the photoresist layer underneath does not have reflows. The final layer of Parylene is two microns thick (cf. section 4.3).
- 3. Inlets and Cantilevers Definition(1):** In order to create openings for the inlet/outlet, protection is needed to protect the parts that we did not want to damage. We used photoresist for this (cf. section 4.4.1).

4. **Inlets and Cantilevers Definition(2):** After the protective photoresist layer is applied, the parylene from the inlet/outlet and around the cantilever is etched. As well as the LS-SiN around the cantilever.
5. **Cantilevers Release:** Once the silicon around the cantilever is accessible, it needs to be etched in order to release the cantilever (cf. section 4.5.2).
6. **Channels emptying:** With the cantilevers released, the last step is to empty the microchannels with the correct solvent in order to use any liquid inside of them (cf. section 4.6).

## 4.2 Design of the channels and inlets via Photolithography

### 4.2.1 Design

The first step is to do a photolithography in order to design the future channels as well as the inlet and outlet (cf. Figure 1).

The first mask can be seen on Figure 12, the bigger squares at the bottom represent the inlet/outlet leading to the microchannels via narrowing channels.

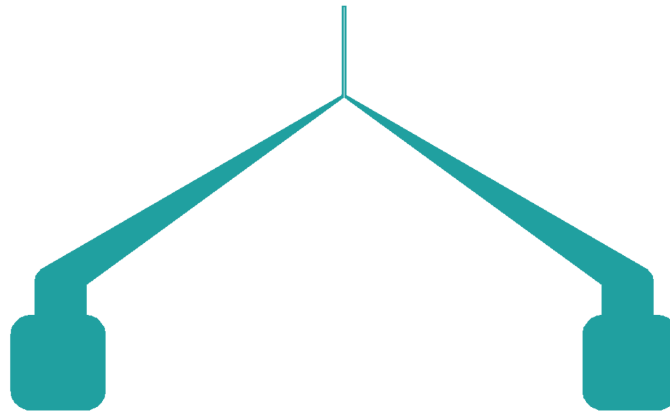


Figure 12: Photolithography First Mask

This photolithography was done by Direct Laser Writing, it means that after being completely coated with photoresist, the wafer's area where we wanted the photoresist to go away (around the blue shape on Figure 12) were exposed to UV not by a classic shadow-mask but by a really precise laser that will locally depolymerize the photoresist. After this exposition, the wafer is washed through a developer bath where the depolymerized photoresist will be washed away revealing the desired pattern.

The photoresist used is the AZ9221 (which is the AZ9260 diluted) with a thickness of 5 microns and a final exposition dose of  $400\text{mJ}/\text{cm}^2$  (cf. next section).

### 4.2.2 Dosetests

After the first photolithography, we noticed that the results were not perfect, as seen on Figure 13, the design is  $10\mu\text{m}$  wide lines with  $10\mu\text{m}$  between them. The over exposition reduced these lines to less than  $7\mu\text{m}$ . This first photolithography was done with exposition parameters of silicon and in our case we have a  $100\text{nm}$  thick LS-SiN layer on top that influenced the reflectivity of the laser during the exposition hence the wrong result.

In order to find the correct exposition needed to obtain something like the Figure 15b instead of this Figure 14a we had to do a dosetest.

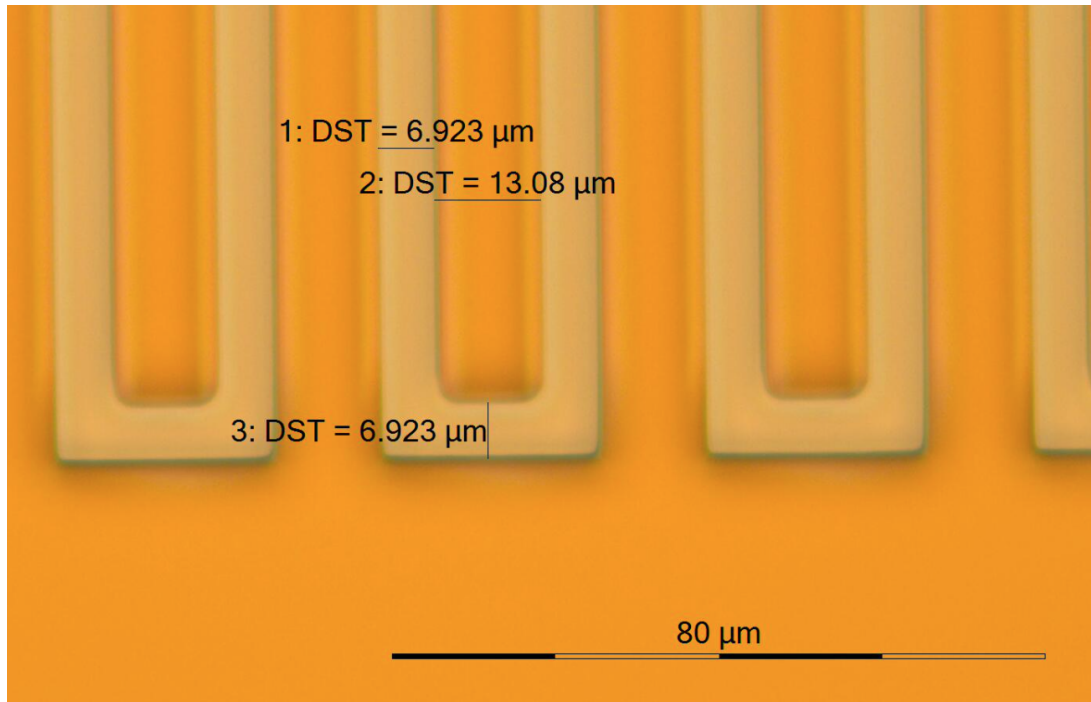


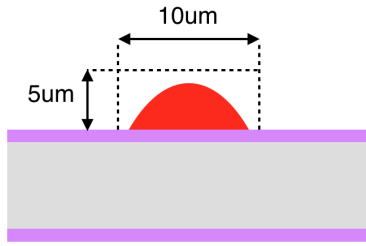
Figure 13: First Photolithography with a  $800\text{mJ}/\text{cm}^2$  dose

It consists in exposing the same design with a range of doses in order to select the one where the result is coherent with the design.

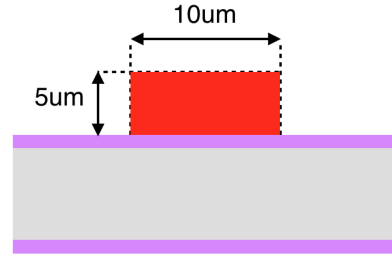
For this dose-test, I patterned  $10\mu\text{m}$  wide lines with doses from  $200\text{mJ}/\text{cm}^2$  to  $450\text{mJ}/\text{cm}^2$  with  $50\text{mJ}/\text{cm}^2$  steps (cf. Figures 14c, 14d, 14e, 14f, 14g, 14h).

After comparison of the different results, a dose of  $400\text{mJ}/\text{cm}^2$  gives the best result with a good  $10\mu\text{m}$  wide final photoresist pattern.

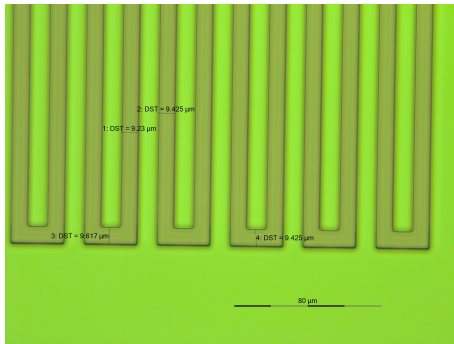
After this, all of the photolithographies were done with a  $400\text{mJ}/\text{cm}^2$  dose.



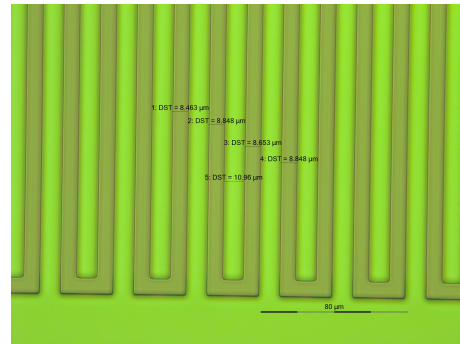
(a)  $800\text{mJ}/\text{cm}^2$



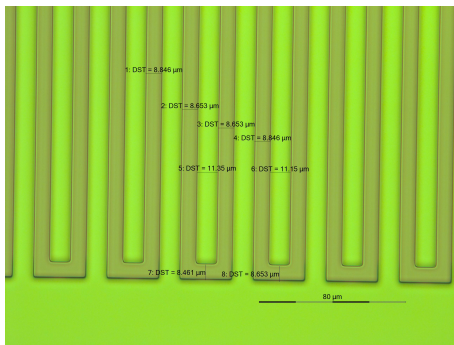
(b)  $400\text{mJ}/\text{cm}^2$



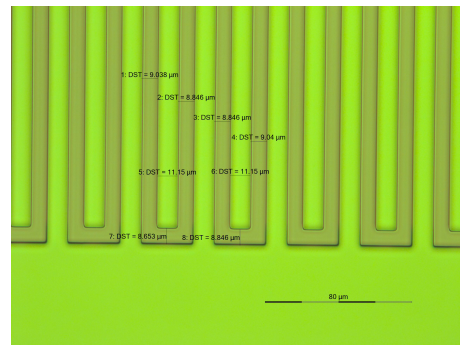
(c)  $200\text{mJ}/\text{cm}^2$



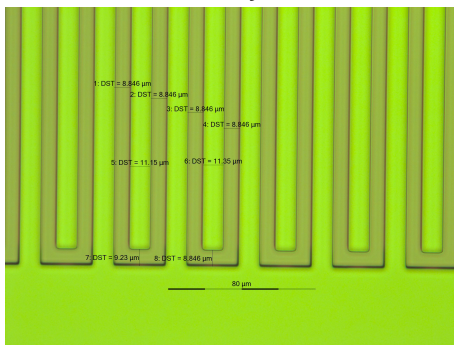
(d)  $250\text{mJ}/\text{cm}^2$



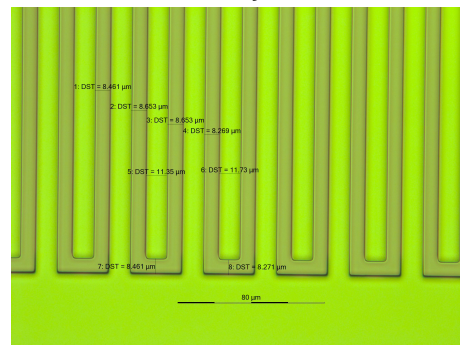
(e)  $300\text{mJ}/\text{cm}^2$



(f)  $350\text{mJ}/\text{cm}^2$



(g)  $400\text{mJ}/\text{cm}^2$



(h)  $450\text{mJ}/\text{cm}^2$

Figure 14: Cross-section representation of the results obtained with different doses:  
**(a)**  $800\text{mJ}/\text{cm}^2$  **(b)**  $400\text{mJ}/\text{cm}^2$   
**(c)** **(d)** **(e)** **(f)** **(g)** **(h)** Pictures of the dosetest results

### 4.3 Encapsulation of the microchannels with parylene C

As explained in the previous section, the parylene deposition is done at room temperature in a low pressure environment (around  $0.001\text{atm}$ ).

To make sure of the correct thickness of parylene needed to ensure a good hermiticity, a substrate of photoresist covered with two microns of parylene was immersed into a solvent bath.

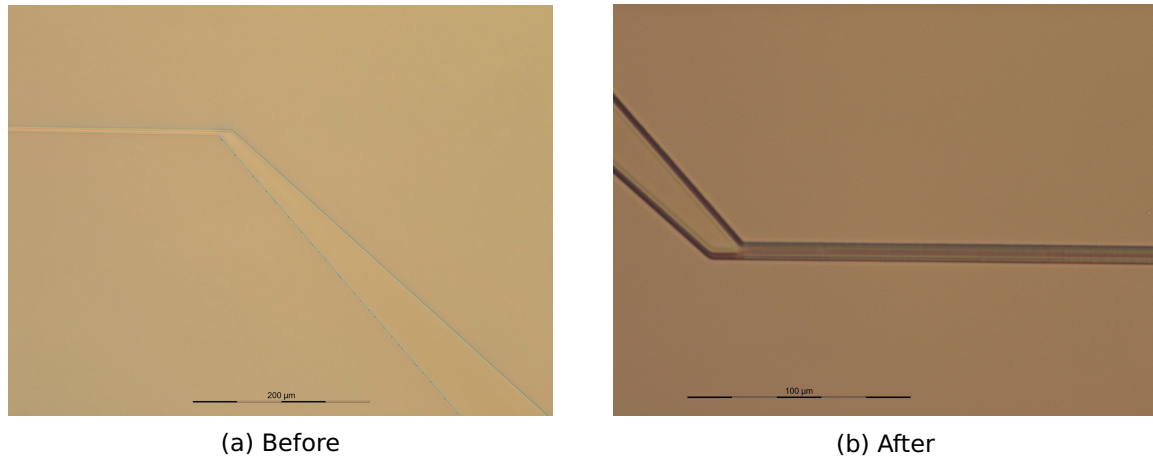


Figure 15: Photoresist covered with a  $2\mu\text{m}$  thick parylene layer  
**(a)** Before **(b)** After 90 seconds in a UFT 1165 Remover solution at room temperature

On the Figure 16, we can see that the solvent did not penetrate the parylene layer and did not damage the photoresist underneath.

It means that a two microns thick parylene layer is enough to ensure a good hermiticity in the channels.

### 4.4 Opening of the inlets

#### 4.4.1 Second lithography aligned on the initial design

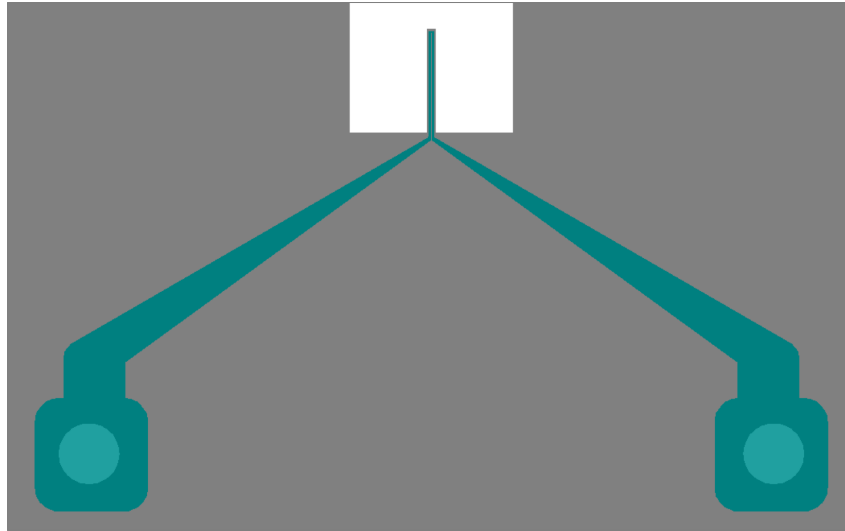
Once the parylene is deposited, the next step is to make the openings for the inlet/outlet at the end of the channels and draw the final shape of the cantilevers.

In order to do that, as it will necessitate some etching, we need to protect the rest of the design. In our case, a thick layer of photoresist was put to resist the etching.

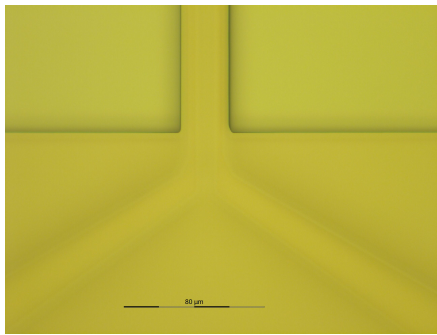
We coated the wafer with a mask as seen on Figure 16a aligned on the first one visible underneath. Everything is covered except two holes of  $300\mu\text{m}$  diameters in each inlet/outlet and a rectangular shape around the cantilever.

On Figures 16b and 16c, we can see the pattern covered with the second layer of photoresist. We used the same thickness and type as for the first photolithography ( $5\mu\text{mAZ9221}$ ).

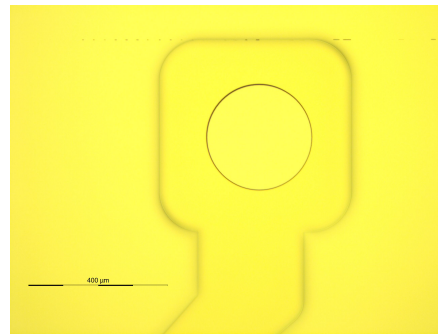
The second layer is well aligned as we can see the inlet well placed and the cantilever being well defined around the microchannels.



(a) Photolithography second mask



(b) Base of the cantilever covered with the protective photoresist



(c) Inlet of the microchannel covered with the protective photoresist

Figure 16: Second photolithorgraphy

**(a)** Second mask

**(b)** Zoom at the base of the microchannels

**(c)** Zoom on an inlet/outlet

#### 4.4.2 Dry etching of the parylene and the LS-SiN

We are now at the fourth step of Figure's 11 process. The objective is to etch the parylene inside the inlet/outlet, and etch the Parylene/LS-SiN layer around the cantilevers.

The first part was to etch the Parylene by dry etching, the technique used is described on Figure 17, the feed gas used is dioxygen ( $O_2$ ). The principal problem is that dioxygen plasma etching also etches photoresist, I had to make sure that the time needed to etch the two microns of parylene would not completely etch the five microns thick photoresist protective layer.

The experiments described in the following Table show that one minute in the machine should be enough to etch the two microns of parylene, and around 2.5 microns of photore-sist will be left in the protective layer.

Material thickness	Initial	Aft.15secs	Aft.30secs	Aft.1min	Etching Rate
Parylene	$1.83\mu m$	-	$0.72\mu m$	-	$2.22\mu m/min$
Parylene	$0.72\mu m$	$0.14\mu m$	-	-	$2.32\mu m/min$
Photoresist	$4.92\mu m$	-	-	$2.53\mu m$	$2.38\mu m/min$
Photoresist	$4.92\mu m$	-	$1.31\mu m$	-	$2.43\mu m/min$



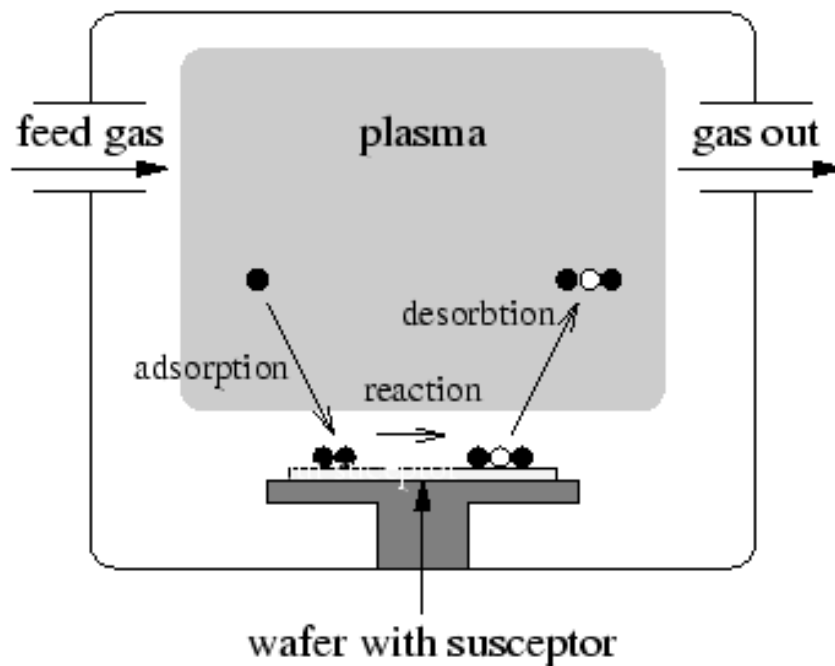


Figure 17: Dry Etching Process

To make sure that the inlet/outlet was correctly opened, I used the same technique as the porosity check. I dipped the etched wafer inside a solvent bath to see if the photoresist was reached.

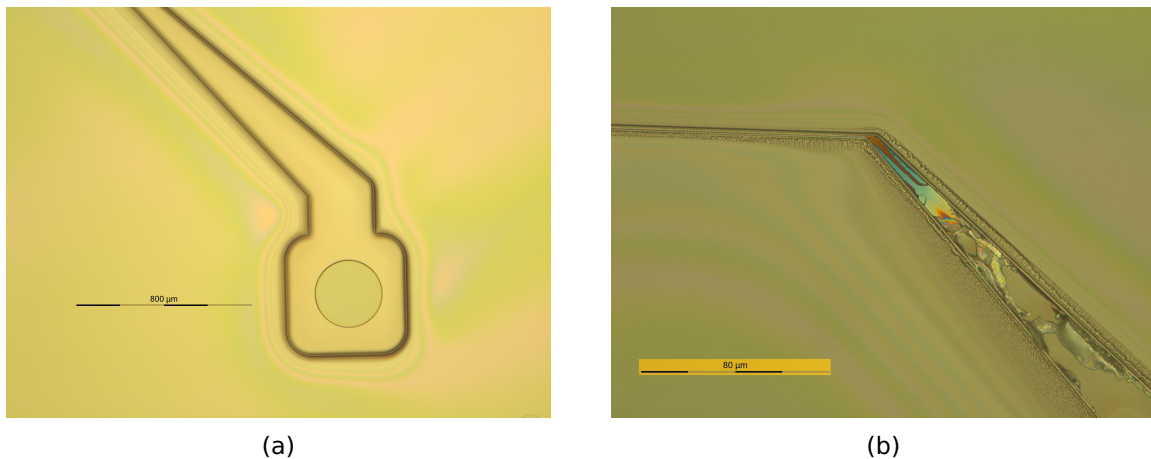


Figure 18: Before and after UFT solvent bath  
**(a)** Before **(b)** After 90 seconds in UFT at Room Temperature

We can see on Figure 18b that even if the photo-resist is not perfectly dissolved, it was reached by the solvent thus confirming the correct etching of the inlet/outlet parylene layer.

For the LS-SiN etching, the same machine was used but the gas for the reaction inside the chamber was different, it is a mix of Fluoroform ( $\text{CHF}_3$ ) and Sulfur hexafluoride ( $\text{SF}_6$ ) that allow the etching of LS-SiN. As only  $100\text{nm}$  need to be etched and the selectivity to photoresist is 2:1, there is no risk to remove all the photoresist protective layer. The etching rate is between  $160$  and  $220\text{nm/min}$  depending on the area of LS-SiN being exposed. To

be sure of the complete etching, the process ran for one minute.

For both recipes, the substrate-holder cools the wafer to 10°C ensuring no damages onto the photoresist.

## 4.5 Cantilever release, etching of the Silicon

Once the Parylene and LS-SiN are removed, the final step is to etch the silicon underneath the hanging cantilevers in order to release them.

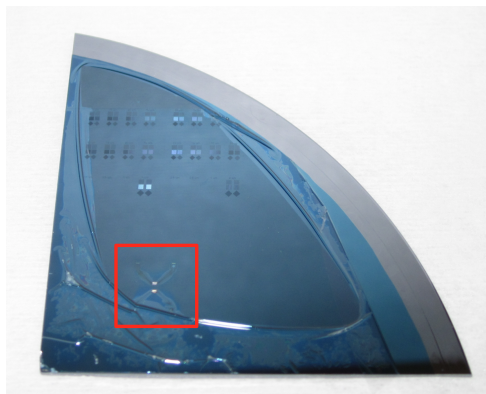
Two different techniques were tested, first via wet etching, the second one via dry etching.

### 4.5.1 Wet etching

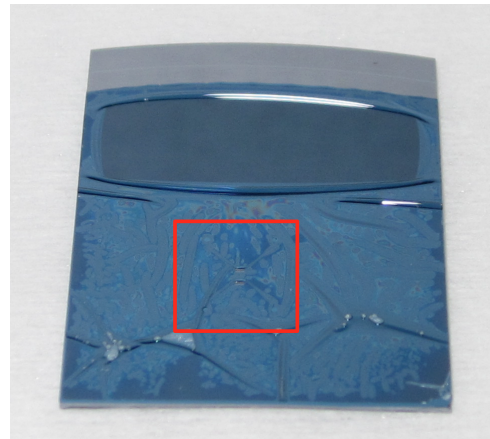
The chemical solution chosen for wet etching is Potassium Hydroxide (KOH), with a 40% concentration, at 60°C, its etching rate is 20 $\mu\text{m/hr}$ .

As the cantilever is 40 $\mu\text{m}$  wide, and we want to make sure of a full release, we planned to let them for three hours as an over-etch does not jeopardize anything on the design

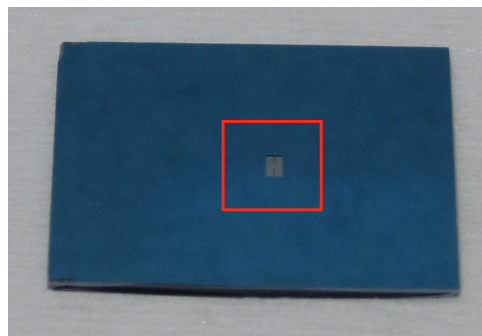
Unfortunately, as you can see on Figure 19, after only one hour, the Parylene/Photoresist layered began to peel from the substrate.



(a) The red square represents where the patterned cantilevers should be visible



(b) The red square represents where the patterned cantilevers should be visible



(c) On this chip, the Parylene layer was completely peeled off, the little silver squares represent the LS-SiN etched

Figure 19: Chips with cantilevers patterned after 30 mins in 40% KOH solution at 60°C

After these results, we decided to stop with wet etching and to opt for dry etching.



#### 4.5.2 Dry etching

The dry etching of silicon was performed in a Deep Reactive Ion Etching equipment (DRIE) more adapted for silicon and especially to obtain high aspect ratio in it. On Figure 20, you can see at the bottom the substrate holder, cooled with liquid nitrogen beneath the diffusion chamber once again to prevent any reflows of the photoresist.

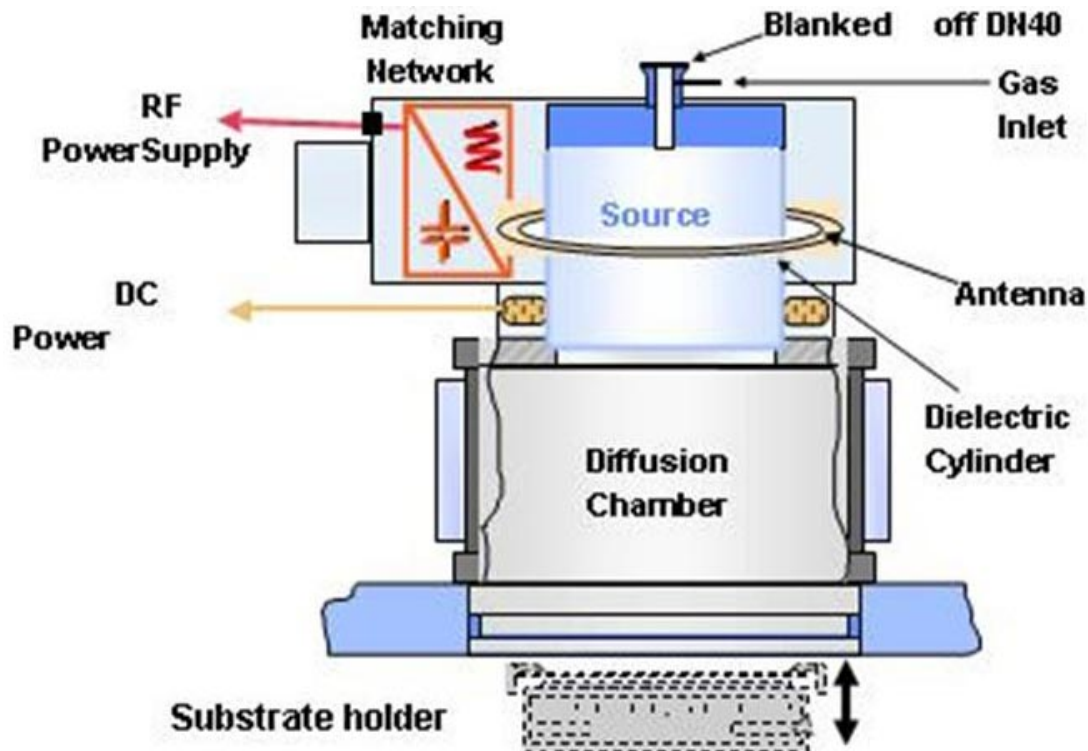


Figure 20: DRIE equipment cross section

The actual step 5 from Figure 11 is actually divided in two different etchings:

- **Anisotropic etching:** This first step consist of digging anisotropically inside the silicon as can be seen on Figure 21. Such an etching is made possible by the alternance of two gases being inserted into the diffusion chamber of the DRIE equipment.

During a first period,  $SF_6$  is injected in order to etch the silicon in every direction and then  $O_2$  is injected to form a protective passivation layer along the walls. By repeating these short steps, the silicon is etched vertically. The difference in the vertical and lateral etching rates make this process possible.

After two minutes, the depth of the trenches was  $15\mu m$  ( $19.5 - 2(\text{Parylene}) - 2.5(\text{PR})$ ) cf. Figure 22.

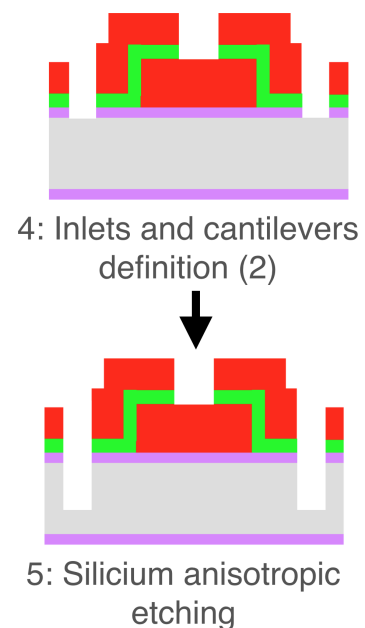


Figure 21: Dry etching: step 1

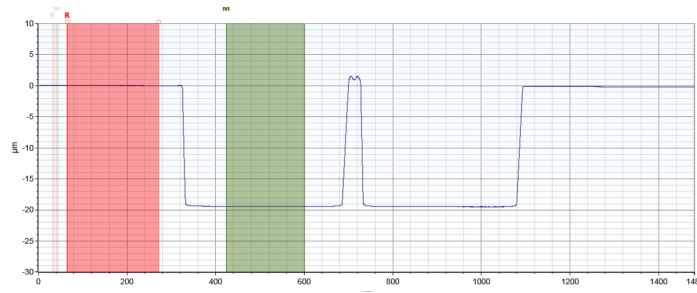


Figure 22: Profiler diagram of a cantilever after anisotropic etching

- **Isotropic etching:** After the anisotropic etching, we etch the silicon isotropically by only injecting  $SF_6$ . It allows us to go beneath the cantilever.

The lateral etching rate of the isotropic etching is  $6\mu m/min$  and we want to release the  $40\mu m$  wide cantilever. As the etching is coming from both sides, the process runs for four minutes to be sure of the complete release.

On Figures 24a and 24b, you can see that the cantilever is completely released. The orange regions indicate a layer of LS-SiN with Parylene on top and they stopped at 25 microns from the side.

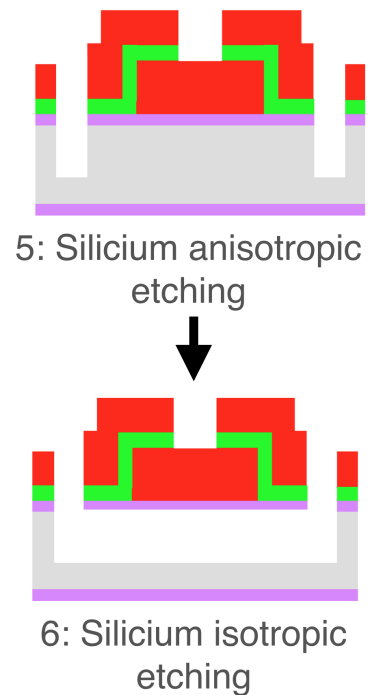
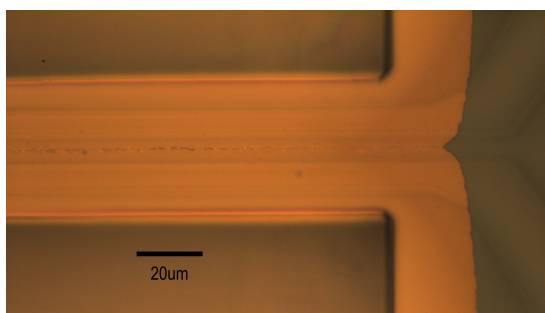
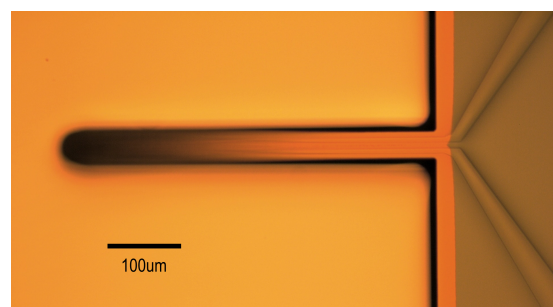


Figure 23: Dry etching: step 2



(a) Released cantilever



(b) Released cantilever

Figure 24: Pictures of the cantilever after 4 minutes of anisotropic etching and 2 minutes of isotropic etching

These Figures also show the fact that the cantilevers are bending a bit upwards at the end (out of focus), as the SiN is Low Stress and the parylene stress free, its origin needs to be studied. From theoretical suppositions, the upward bending should indicate that parylene had a little of extensive stress inside itself.

## 4.6 Emptying the channels, etching of the Photo-Resist

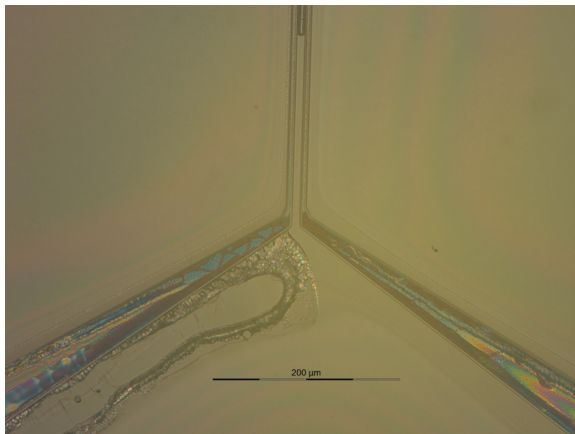
The final step is to empty the channels by removing all of the photoresist inside of them but without damaging the parylene layer.

### 4.6.1 Selection of the best solvent

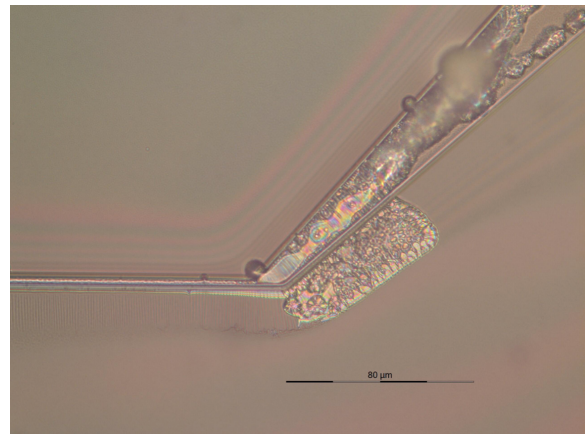
First of all, I had to select the solvent among those available in the laboratory. I had at my disposition:

- Acetone
- SVC-14
- Technistrip
- UFT Remover 1165

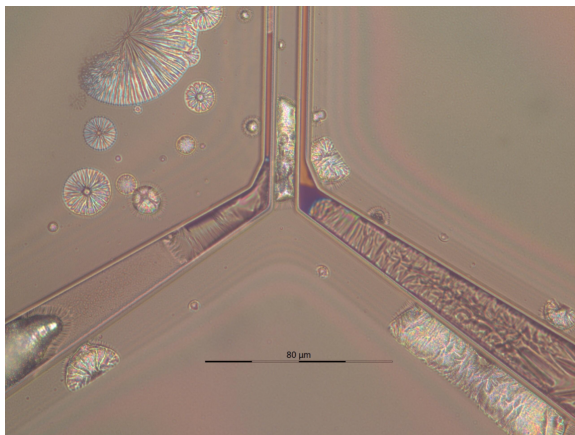
My goal was to dip a patterned substrate inside each of these solvents for one minute at room temperature and see the results.



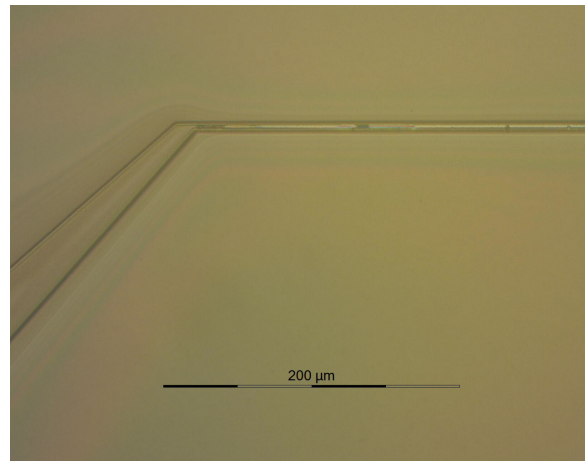
(a) Acetone



(b) SVC-14



(c) Technistrip



(d) UFT Remover 1165

Figure 25: Pictures of microchannels emptied by different solvents for one minute at room temperature

From these pictures only, it is clear that the best solvent is the UFT Remover 1165. It did not damage any of the parylene and the photo resist is almost completely gone.

#### 4.6.2 Determination of the etching parameters

Unfortunately what the Picture 25d shows is not perfect. The channels need to be completely empty in order to be used as we planned to.

Many experiences were needed in order to determine what are the optimum parameters in order to remove all of the photoresist. Three parameters can vary:

1. **Temperature:** I tried to heat a little the solvent solution to 70°C and see if it makes any difference.

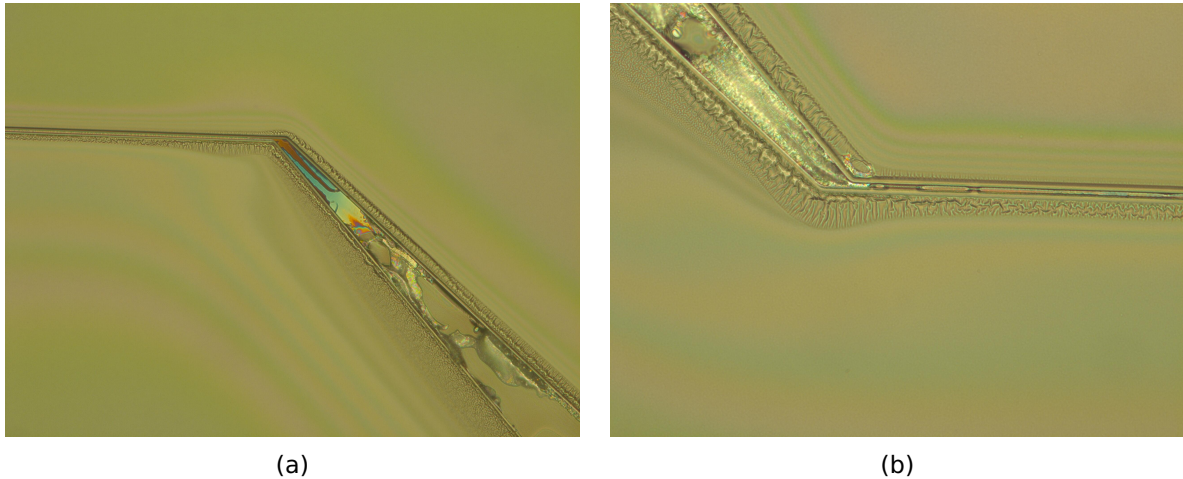


Figure 26: Pictures of microchannels emptied by UFT 1165 for one minute at 70°C

We clearly see that it is not the good solution; there is more photoresist than at room temperature and the channels does not look as well defined as before the bath.

2. **Ultrasound:** The idea is to see if the ultrasounds can help the solvent to reach the microchannels.

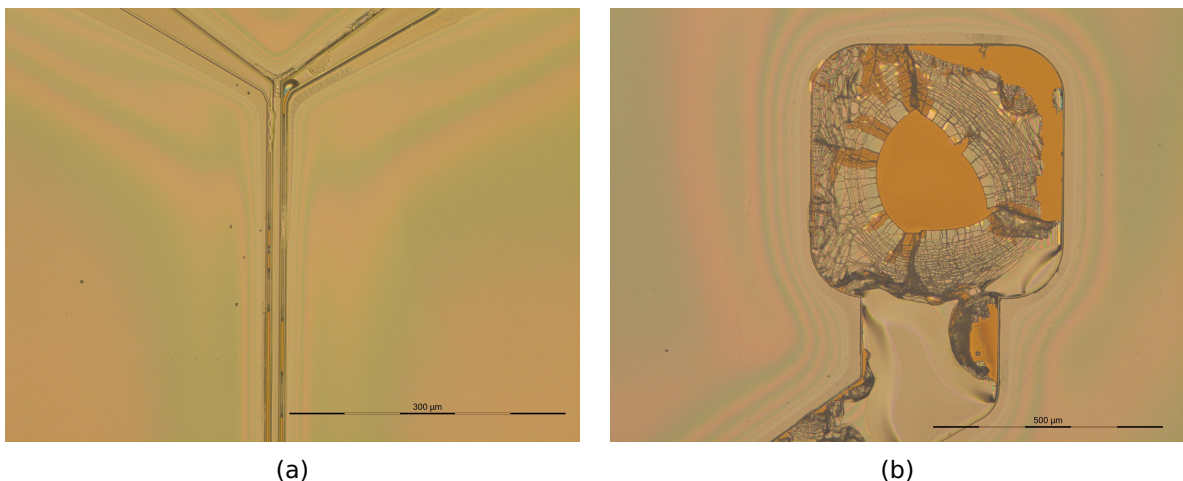
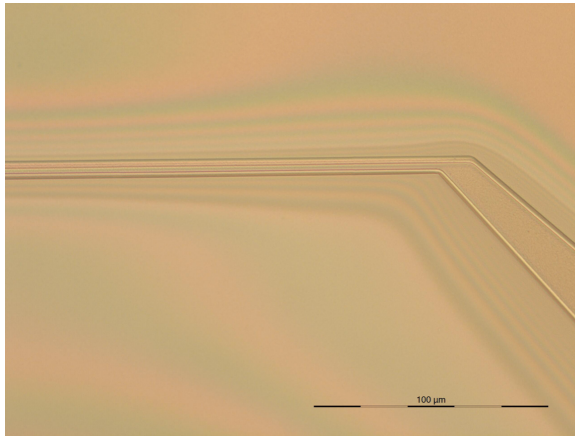


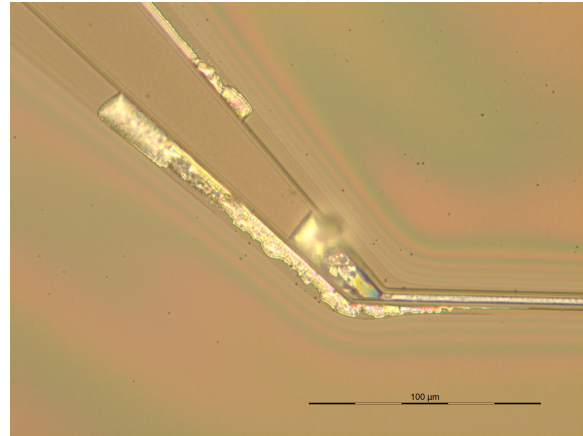
Figure 27: Pictures of microchannels emptied by UFT 1165 for one minute at room temperature with ultrasounds

On Figure 27a, we see that the channels are still not well emptied and it seems like the Parylene begins to unstick from the substrate. On Figure 27b we see that the ultrasounds completely ripped apart the parylene layer. It is definitely not the way to go.





(a) 90 seconds



(b) 120 seconds

Figure 28: Pictures of microchannels emptied by UFT 1165 for one minute at room temperature with ultrasounds

3. **Time:** The final parameter is the time spent in the bath.

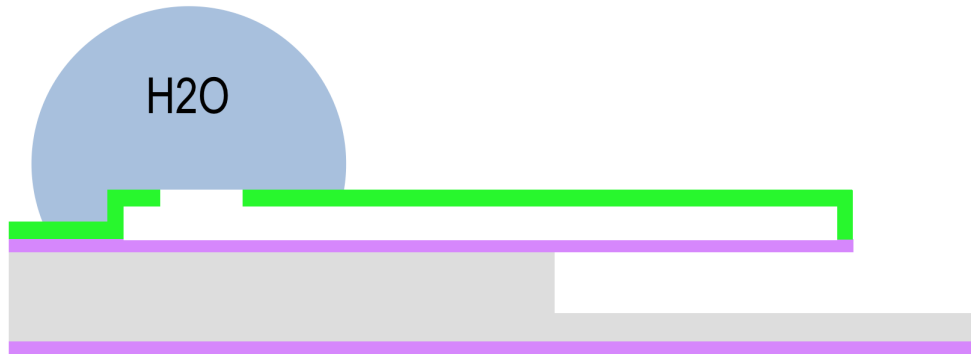
After two new durations, it is clear that after 90 seconds, the channels are well better emptied but 120 seconds is too much and it begins to damage the channels.

After these experiments, all the channels are emptied in a UFT Remover 11165 bath for 90 seconds at room temperature.

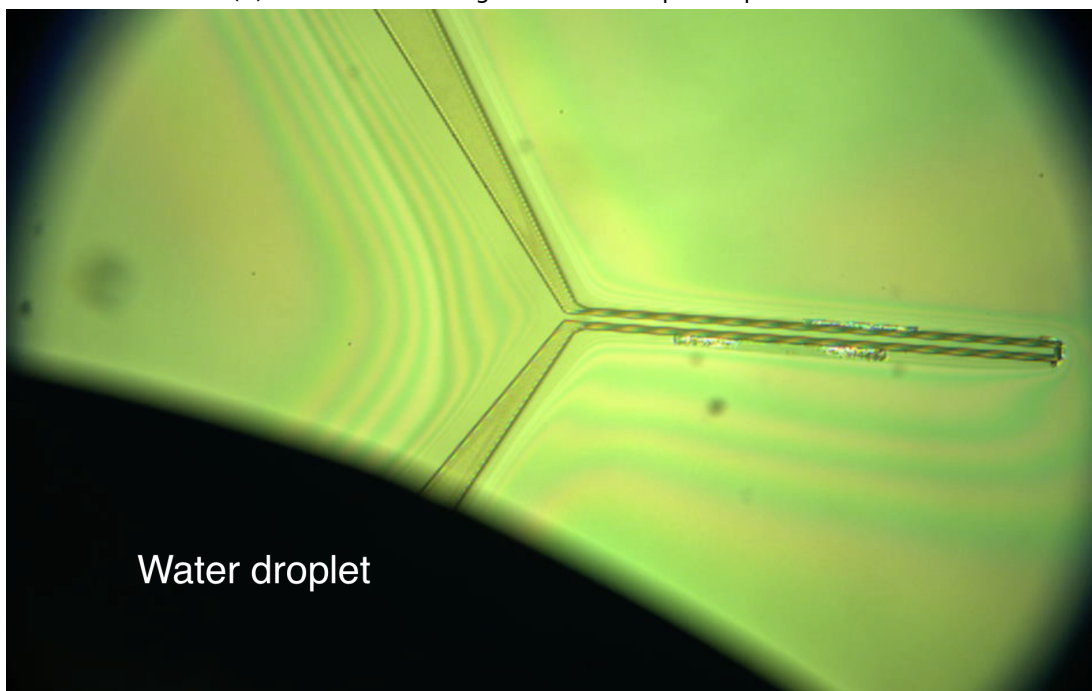
## 5 Liquid circulation test

The last experiment I performed for the project was to see if a liquid could circulate easily inside the created microchannels. To this end, I took a prototype with empty channels but unreleased cantilever and deposited a droplet of water on top of the open inlet.

The idea is to see if the water is attracted inside the channel.



(a) Cross section diagram of the droplet experiment



(b) Picture of the empty cantilever with the droplet of water on top of the inlet

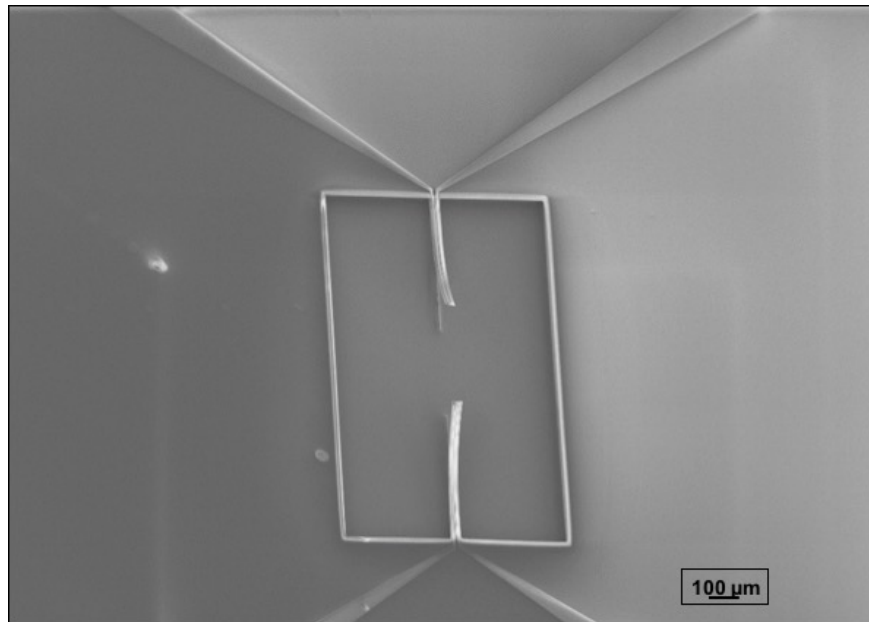
Figure 29: Water droplet experiment

Unfortunately, the Figure 29 shows what happened. The droplet stayed on top of the inlet but none of the liquid went inside the channel. As not obvious on a picture, this experiment was conducted under video recording and the analysis clearly showed none of the liquid inside the channels.

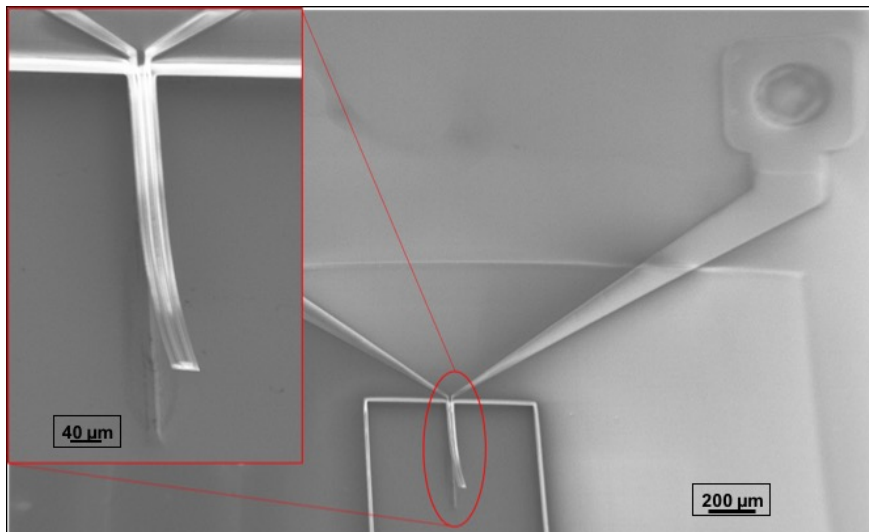
It indicates that the parylene is not hydrophilic enough to attract the liquid, we will need some pressure on the liquid to force the circulation of liquid inside the channels.

## 6 Results

All these steps allowed me to make a completed device, seen on Figure 30.



(a)



(b)

Figure 30: Completed device

On these pictures, two  $500\mu\text{m}$  long,  $40\mu\text{m}$  wide and  $100\text{nm}$  thick cantilevers made of Low Stress Silicon Nitride are facing each other. Each one contains an empty microchannel  $10\mu\text{m}$  wide and  $5\mu\text{m}$  high made by a  $2\mu\text{m}$  high parylene layer .

These microchannels are leading via wider channels to openings used as inlet and outlet to enable the circulation of the liquids needed for the final application of these cantilevers. These openings have a  $300\mu\text{m}$  diameter.

## 7 Conclusion & Outlook

This project's main goal was to determine if it was possible to use the Solid on Liquid technology to fabricate microchannels on top of Low-Stress Silicon Nitride cantilevers. The first advantage of this technology was the fact that the parylene could be deposited on top of a liquid, which would ease the emptying of the channels after being covered by a parylene layer.

The first step was to find a way to pattern the desired channel design on top of the LS-SiN substrate. The results were not as good as envisaged because the hydrophobic treatment chosen, FOTS, was not going well with LS-SiN and further experiments would be needed to determine with precision what could be modified or optimized in order to continue this way.

However, a second approach was followed: the use of photoresist as a sacrificial material to be covered by parylene. This approach shows promising results as I was able to correctly pattern the microchannels as well empty them after the deposition layer of parylene.

In the following steps, a protocol was followed to enable the release of these cantilevers. After protecting the parylene layer with photoresist, several steps of dry etching successfully released the LS-SiN cantilever from its silicon substrate.

In conclusion we can say that even though the actual Solid on Liquid technology was not used in this project, we were able to fabricate suspended cantilevers with empty parylene microchannels on top of it.

This project will be followed up by another one next semester where the same cantilevers will be fabricated but this time with microchannels engineered by low temperature atomic layer deposition to replace the parylene in hope of improved results.



## References

- [1] K. L. O. Ili and R. Garcia. Temperature-dependence of the contact angle of water on graphite, silicon, and gold. *Water*, page 84, 2009.
- [2] S. C. Systems. Parylene conformal coating specifications & properties. pages 1–12, 2013.
- [3] T. Tran, U. Le, A. Sasahara, and M. Tomitori. Water wettability of an ultrathin layer of silicon oxide epitaxially grown on a rutile titanium dioxide (110) surface. (110), 2013.